Research Article

Katarzyna Szwedziak, Żaneta Grzywacz*, Ewa Polańczyk, Sławomir Tomaszewski, Wiktoria Wojtkiewicz

Physicochemical properties of selected herbicidal products containing nicosulfuron as an active ingredient

<https://doi.org/10.1515/chem-2020-0045>

received September 17, 2019; accepted February 16, 2020

Abstract: Nicosulfuron is a herbicide used for plant protection. This paper presents the results of research on the physicochemical properties of three herbicidal preparations containing nicosulfuron as the active substance by measuring and comparing its concentration by the chromatographic method. These preparations are in the form of a concentrated suspension intended for dilution with water, and due to the fact that nicosulfuron contained in the tested preparations is sensitive to water quality, while checking the physicochemical properties of the preparations, a dispersion for soft (pH: 5–7) and hard (pH: 8–9) water was also determined. It is important to note that high content of mineral salts in water may have a negative effect on the biological activity of the substance. In addition, the use of herbicides does not only apply to the use of appropriate dilutions, which are effective and not harmful to the environment. However, it is also important to consider specific farmer and equipment used, so that the preparation is easy to use and does not adversely affect the kit used for spraying. This study has shown that depending on the preparation used based on the amount of active substance 40 g/l, the amount of nicosulfuron varies. The test indicators in the form of pH and density are similar, while during the tested dispersion, differences depending on the tested water were observed (sediments were observed in only one preparation tested). Differences depending on the occurrence of sediments in

the water used, both at 0 and 24 h, are signaling in favor of the soft water. Therefore, it is important to assess the hardness of the water, which in turn guarantees a reduction of the amount of deposits and protection of agricultural equipment used for spraying.

Keywords: chemical substance, nicosulfuron, herbicide, weed killer, plant protection

1 Introduction

The negative impact of weeds on crop and, above all, huge advances in research on their biology and control, in particular with the use of chemical methods, contribute to both the development of a science called herbology and the intensification of herbicide production [1]. Herbicides are classified as plant protection products (PPPs) [2]. To manufacture herbicides, especially herbicides combating weeds in maize cultivation, an active substance called nicosulfuron plays an important role. Exemplary outputs containing nicosulfuron include Nicorn 040 SC, Niko-sulfuron 040 SC and Maksymus 040 SC. These are concentrates of condensed suspensions for water dilution, and nicosulfuron is one of the compounds susceptible to water properties.

Maize (*Zea mays* L.) is easily dominated by weeds [3,4] due to its slow initial growth and low density on a surface unit. Therefore, an effective corn weed management, almost exclusively, is based on the application of herbicides [5].

In general, the production and the use of appropriate PPPs are of great importance, and it particularly concerns the quality of the applied substance as well as its physicochemical properties since manufacturers of PPPs store them for further processing, sometimes for many months. As a result, these substances are exposed to various external factors [6]. The effectiveness of

* Corresponding author: Żaneta Grzywacz, Opole University of Technology Faculty of Production Engineering and Logistics, Stanisława Mikołajczyka 5, 45-271 Opole, Poland, e-mail: z.grzywacz@po.opole.pl

Katarzyna Szwedziak, Ewa Polańczyk: Opole University of Technology Faculty of Production Engineering and Logistics, Stanisława Mikołajczyka 5, 45-271 Opole, Poland

Sławomir Tomaszewski: Pestila Ltd. Wolbórz near Tomaszów Mazowiecki, Poland

herbicides used in the form of aqueous solutions also considerably depends on the applied usable solution. If the condensation of mineral salts in water is too high, the biological activity of the substance may be impaired [7,8]. Thus, the interplay between chemical components of water and the emulsion quality may be of key importance [9]. Therefore, PPPs require a more vivid insight into the assessment of both chemical compounds and water used for their production, which may contribute to the emergence of more effective herbicidal products development [10]. The effectiveness of these agents can be determined based on physicochemical tests. In addition, it should be remembered that the use of herbicides is not only about using appropriate dilutions, which are effective and do not poison the environment. However, it is also important to consider specific farmer and the equipment used, so that the preparation is easy to use and does not adversely affect the kit used for spraying, thus hindering the application of the treatment.

2 Experimental part

2.1 The aim and scope of the study

The aim of this study is to determine, evaluate and compare the physicochemical properties of the three selected preparations: Nicorn 040 SC, Nikosulfuron 040 SC and Maksymus 040 SC. The scope of this study is based on the concentration of the active ingredient used in the production of herbicides and determining the dispersion for two types of water used: soft water (pH: 5–7) and hard water (pH: 8–9). This study focused on monitoring of the active substance called nicosulfuron.

2.2 The material and methods

The study was carried out on three herbicidal products containing nicosulfuron as their active ingredient (Nicorn 040 SC, Nikosulfuron 040 SC and Maksymus 040 SC), which is responsible to control weeds such as cockspur, field pansy, cleavers, black bindweed and lamb’s quarters.

The tested preparations are herbicides in the form of a concentrated suspension with systemic activity. The assimilation mainly involves leaves. After penetrating into a plant, they inhibit its growth and development.

Weed growth is limited shortly after the application of the agent. However, the final decay occurs 20–25 days after the application. According to the manufacturer, the highest effectiveness of the tested preparations can be observed on young, intensively growing weeds, at the stage of 2–4 leaves.

Since the aim of this study is to determine the physicochemical properties of the selected preparations and to measure the concentration of the active ingredient called nicosulfuron, the following analyses were performed: direct pH determination, pH determination in 1% emulsion, determination of the sediment, determination of volume of its upper cream and oil, determination of dispersion for the two types of the applied water and determination of the content of nicosulfuron in the products. The tests were conducted in accordance with the good laboratory practices relying on the international methods. For this purpose, a model solution and a test solution are prepared. A chromatographic method is applied together with the liquid chromatography and Agilent Technologies 1260 Infinity device (Table 1). The active ingredient (nicosulfuron) content in the products is calculated according to the following equation:

Z = (A_bad * m_wz * P * d) / (A_wz * M), (1)

where Z is the concentration of nicosulfuron in the product (g/l), A_{bad} is the surface area of nicosulfuron in the chromatogram of the tested solution, A_{bad} is the surface area of the nicosulfuron peak on the chromatogram of the model solution, m_{wz} is the weighted amount

Table 1: Characteristics of the LC chromatography instrument [source: own research]

Chromatographic system: HPLC apparatus	
DAD detector	260 nm wavelength
HPLC column	Zorbax eclipse XDB-C18, 150 mm × 4.6 mm × 5 µm or equivalent
Flow	1.2 mL/min
Column temperature	30°C
Injection volume	PTFE 0.45 µm
Syringe filters	PTFE 0.45 µm
Analysis time	10 min
Medium	10% tetrahydrofuran solution in acetonitrile
Mobile phase	A: 0.1% orthophosphoric acid, B: acetonitrile, A : B: 70 : 30

of the model (mg), M is the weighted amount of the technical product (g/l), P is the purity of the model substance nicosulfuron (%) and d is the density of the technical product (g/l).

The mean value of at least two parallel determinations, not different from each other in more than 2%, is assumed to be the final result.

Subsequently, a test involving a potentiometric measurement of product pH with the use of a pH meter equipped with a composite electrode is performed. The pH was measured for a 1% suspension of the formulation and directly in the formulation at 20°C. To indicate the stability of a suspoemulsion, a basic laboratory glass and water A and D are used for every trial. The pH of water A (soft) is 5–7 and that of water D (hard) is in the range of 8–9. Two graduated cylinders (250 ml) are filled with 240 ml of standard water of $23 \pm 2^\circ\text{C}$ temperature. The preparation (5 g) is added with a pipette dropwise to each cylinder, and then, they are made up with standard water up to 250 ml. The cylinder is subjected to rotation of 30 times by 180° , and its content is used to perform sedimentation and creaming tests, while the content of the second cylinder is treated in a re-dispersion test. Next, the dispersions and all traces of sediment, cream or oil are observed. After the dispersion is formed, 100 ml of the solution from the first cylinder is transferred to an emulsion tube. It is plugged with a stopper and left at room temperature for 30 min. Then, the tube is illuminated with a lamp, adjusting the position of the light angle for the optimal view of the interface, and the volume of the sediment is recorded with an accuracy of ± 0.05 ml. Immediately after formation of the dispersion, the emulsion tube is filled up to a volume of about 1 mm. Then, the tube is plugged with a rubber stopper with a ventilation tube. The tube is then reversed and left in the position at room temperature for 30 min. The volume of the formed cream or oil is recorded and the total volume of the tube is determined, and the measured volume receives correction according to the following equation:

$$F = \frac{100}{V_0}, \quad (2)$$

where F is the correction coefficient, which should be applied to the measured volume of cream or oil and V_0 is the total volume of the emulsion tube.

After the initial dispersion, the second cylinder is left uninterrupted for 24 h at room temperature, and the aforementioned steps are repeated.

Ethical approval: The conducted research is not related to either human or animal use.

Table 2: Descriptive statistics for the results of individual tested products [source: own research]

Trade name of the product	Tested variable	N	Average	Confidence -95%	Confidence +95%	Median	Minimum	Maximum	Variance	Standard deviation	Standard error
Nicorn 040 SC	Concentration	16	41.14	40.27	42.02	41.45	37.6	43.2	2.676	1.64	0.41
	pH	16	4.86	4.8	4.91	4.9	4.6	5	0.012	0.11	0.03
	Density	16	0.98			0.98	0.98	0.98	0	0	0
Nikosulfuron 040 SC	Concentration	3	41	37.24	44.76	40.5	39.8	42.7	2.29	1.51	0.87
	pH	3	4.97	4.82	5.11	5	4.9	5	0.003	0.06	0.03
	Density	3	0.98			0.98	0.98	0.98	0	0	0
Maksymus 040 SC	Concentration	3	41.43	40.92	41.95	41.5	41.2	41.6	0.043	0.21	0.12
	pH	3	4.87	4.72	5.01	4.9	4.8	4.9	0.003	0.06	0.03
	Density	3	0.98			0.98	0.98	0.98	0	0	0

2.3 The analysis and discussion of results

The findings raised in the course of this study enable to accomplish basic descriptive statistics for pH, pH for 1% suspension, density and concentration of nicosulfuron in the tested products (Table 2). Figure 1 illustrates and compares the concentration of the active ingredient, which should be equal to 40 g/l according to the manufacturer's specifications.

Figure 1: Concentration of the active ingredient in the products [own source].

The obtained results enable to assume that the nicosulfuron quantity is different for all the tested preparations. An extraordinary wide range of the substance content can be observed in case of the Nikosulfuron 040 SC product, which is subjected to triple trial, but the variance is 2.290 and the confidence interval is within 37.42–44.76 g/l. Similar results were obtained by Swedziak, Tukiendorf and others while examining the physicochemical properties of azoxystrobin. The tested indicators, such as the content of the active substance, were close to the assumptions in the scope of accepted standards. The average metazachlor content varied depending on the production batch, within the norms [11]. The tested parameters in the form of pH and density approximate for all the tested products according to the standards they demonstrate the lack of negative impact on the tested plants [12]. However, the differences are observed with relation to the trace amounts of sediments, which are only recorded in the product called Nicorn 040 SC. For water A at 0 h, eight cases are observed, whereas for water D, 15 cases are observed. Moreover, after 24 h, the sediment precipitates only in two cases with the use of water A compared to five observed cases with the use of water D (Figure 2 and Table 3).

The presence of sludge may affect nozzle clogging. Oil and sludge are not allowed to be introduced as these would disqualify mixtures by impairing ability to perform the procedure [13].

Figure 2: Amount of trace sediments for the Nicorn 040 SC product depending on the pH of the applied water [own source].

3 Summary

Based on the achieved results concerning selected quality parameters in products applied for weed elimination in maize cultivation, where nicosulfuron is the active substance, it is possible to delineate the following conclusions.

Despite the manufacturer's specification concerning the active ingredient quantity equal to 40 g/l, the amount of nicosulfuron varies depending on the product used. A particularly wide range of the substance content can be observed in Nikosulfuron 040 SC, where the variance equals 2.290 and the confidence interval lays within 37.42–44.76 g/l.

The tested indicators in the form of pH and density approximate in all the tested products. An acceptable range of pH is 4–9, which is known from phytotoxicity experiments that it does not cause negative effects on test plants.

Cream or oil formation is not observed during dispersion assessment, and sediments are indicated as trace amounts.

In the course of the dispersion analysis, differences related to the tested water (A or D) as well as to the commercial product used for the study can be identified. The only product with observed occurrence of sediments is Nicorn 040 SC. However, the differences in the occurrence of sediments depending on the water, both in the 0 h and after 24 h, are in favor of water A.

Table 3: Determination of sediment, upper cream and oil volume at hour 0 and after 24 h for all tested products [source: own research]

Trade name of the product	Test of dispersion using water A						Test of dispersion using water D					
	At hour 0			After 24 h			At hour 0			After 24 h		
	Cream	Oil	Sediment	Cream	Oil	Sediment	Cream	Oil	Sediment	Cream	Oil	Sediment
Nikosulfuron 040 SC	0	0	0	0	0	0	0	0	0	0	0	0
Nicorn 040 SC	0	0	8	0	0	2	0	0	15	0	0	5
Maksymus 040 SC	0	0	0	0	0	0	0	0	0	0	0	0

When choosing Nicorn 040 SC to protect agricultural equipment used for spraying the solution, it is important to determine hardness of the water because the use of soft water will result in a reduction in the amount of deposits.

Conflict of interest: Authors declare no conflict of interest.

References

- [1] Woźnica Z. *Herbologia*. Poznań: Powszechne Wydawnictwo Rolnicze i Leśne Sp. z o.o.; 2012. (in Polish).
- [2] <http://www.pcs.agriculture.gov.ie/plantprotectionproducts/>, access online 01.2019.
- [3] Idziak R, Woźnica Z. Effect of nitrogen fertilizers and oil adjuvants on nicosulfuron efficacy. *Turk J Field Crops*. 2013;18:174–8.
- [4] Sulewska H, Koziara W, Śmiatacz K, Szymańska G, Panasiewicz K. Efficacy of selected herbicides in weed control of maize. *Fragm Agron*. 2012;29(3):144–51.
- [5] Woźnica Z, Idziak R. *Fragm*. Wpływ obniżonych dawek herbicydów stosowanych z adiuwantami w różnych terminach na zachwaszczenie i plonowanie kukurydzy. *Agron*. 2015;32(2):111–8. (in Polish).
- [6] Szwedziak K, Grzywacz Ż, Biłos Ł, Tomaszewski S. Physicochemical properties of containing azoxystrobin AZS-GEN 250 SC after 2 years of storage/Właściwości fizykochemiczne preparatu AZS-GEN 250 zawierającego azoksystorbinę po dwóch latach przechowywania. *Przemysł Chemiczny*. 2019;98(3):372–6. doi: 10.15199/62.2019.3.4.
- [7] Ratajkiewicz H. Skuteczność wybranych fungicydów w ochronie róż przed moczniakiem prawdziwym (*Sphaerotheca pannosa* var. *Rosae*) w zależności od ilości cieczy użytkowej oraz jakości wody użytej do jej przygotowania. *Hortorum Cultus*. 2003;2(2):109–14. (in Polish).
- [8] Woźnica Z. Wpływ związków mineralnych występujących w wodzie na fitotoksyczność soli dwuetanoloaminowej 2,4-D. *Rocz AR w Poznaniu*. 1990;203:42. (in Polish).
- [9] Linder PL. Effect of water in agricultural emulsions pesticide chemistry vol. 5 herbicides fungicides, formulation chemistry. New York, London, Paris: Gordon and Breach Science Publishers; p. 453–70.
- [10] Hollomon DW. Fungicide resistance: facing the challenge – a review. *Plant Protect Sci*. 2015;51:170–6.
- [11] Szwedziak K, Tukiendorf M, Grzywacz Ż, Tomaszewski S, Wojtkiewicz W. Ocena fizyczno-chemicznych właściwości preparatu Metazachlor 500 SC o działaniu chwastobójczym zawierającego metazachlor jako substancję czynną/ Physicochemical properties of a commercial herbicidal product containing metazachlor as an active substance. *Przemysł Chemiczny*. 2019;98(7):1113–7. doi: 10.15199/62.2019.7.16.
- [12] Sobiech Ł, Idziak R, Skrzypczak G. Wpływ pH i napięcia powierzchniowego cieczy opryskowej na skuteczność działania mezotrionu. *Przemysł Chemiczny*. 2018;97(8):1325–7. (in Polish).
- [13] Pruszyński S, Wachowiak M, Stobiecki M. Dobra praktyka ochrony roślin, Zasady mieszania i łącznego stosowania agrochemikaliów. 2013. (in Polish).